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IS 4332-7 (1973): Methods of test for stabilized soils,  
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*Indian Standard*

**METHODS OF TEST FOR STABILIZED SOILS**

**PART VII DETERMINATION OF CEMENT CONTENT OF  
CEMENT STABILIZED SOILS**

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**BUREAU OF INDIAN STANDARDS**  
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# Indian Standard

## METHODS OF TEST FOR STABILIZED SOILS

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**AMENDMENT NO. 1    APRIL 1983**  
**TO**  
**IS 4332 ( PART 7 ) – 1973 · METHODS OF TEST FOR**  
**STABILIZED SOILS**

**PART VII   DETERMINATION OF CEMENT CONTEST OF CEMENT**  
**STABILIZED SOILS**

**Alterations**

( *Page 4, clause. 2.10, line 2* ) – Substitute 'IS : 460 ( Part I ) – 1978†' for 'IS : 460 1962†'.

( *Page 4, clause 3.0, line 2* ) – Substitute 'IS : 1070 - 1977‡' for 'IS : 1070 - 1960‡'.

( *Page 4, foot-notes with '†' and '‡' marks* ) – Substitute the following for the existing:

†Specification for test sieves : Part I wire cloth test sieves (*second revision* ).

‡Specification for water for general laboratory use ( *second revision* ).

( BDC 23 )

## *Indian Standard*

### METHODS OF TEST FOR STABILIZED SOILS

#### **PART VII DETERMINATION OF CEMENT CONTENT OF CEMENT STABILIZED SOILS**

#### **0. FOREWORD**

**0.1** This Indian Standard ( Part VII ) was adopted by the Indian Standards Institution on 16 February 1973, after the draft finalized by the Soil Engineering Sectional Committee had been approved by the Civil Engineering Division Council.

**0.2** Soil stabilization is the chemical or mechanical treatment designed to increase or maintain the stability of mass of soil or otherwise to improve its engineering properties. There are several methods of stabilization and these may be broadly classified on the basis of treatment given to the soil ( for example, dewatering and compaction ), process involved ( for example, thermal and electrical ), and additives employed ( for example, asphalt and cement ). The choice of a particular method depends on the characteristics of the problem on hand and on the nature of soil type encountered. For studying in the laboratory, the methods and effects of stabilization, certain standard methods of test for the evaluation of properties of stabilized soils and their analysis are required. The required standards on methods of test for stabilized soils are being published in parts and this part lays down the method of test for determining cement content of cement stabilized soils.

**0.3** In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by basing the standard on the following publications:

ASTM : D806-1965 Method of test for cement content of soil-cement mixtures. American Society for Testing and Materials.

DEWAN ( RL ). The rapid estimation of cement content in mortar and concrete. Indian Concrete Journal. April 1959.



**IS : 4332 ( Part VII ) - 1973**

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960\*.

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**1. SCOPE**

**1.1** This standard ( Part VII ) lays down the method of test for determining cement content of cement stabilized soils.

**2. APPARATUS**

**2.1 Analytical Balance** — sensitive to 0.001 g.

**2.2 Glass Beakers** — three of 250-ml capacity and two of 600-ml capacity.

**2.3 Glass Funnel** — 6 cm diameter.

**2.4 Burette** — one, 50 ml.

**2.5 Pipette** — 25 ml.

**2.6 Conical Flasks** — two of 250-ml capacity.

**2.7 Filter Papers** — Whatman No. 41 and 42 or equivalent.

**2.8 Heating Equipment**

**2.9 Drying Oven**

**2.10 Sieves** — 40-mm, 20-mm, 10-mm, 4.75-mm and 425-micron IS Sieves conforming to the requirements of IS : 460-1962†.

**3. REAGENTS**

**3.0 Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1960‡ ) shall be used in tests.

**NOTE** — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

**3.1 Potassium Permanganate Solution** — N/10; dissolve 3.2 to 3.25 g of potassium permanganate on a watch-glass and dissolve in one litre of distilled water. Heat the solution to boiling, and allow the solution to cool to room temperature. Filter the solution through a funnel containing a plug of purified glass wool. The solution should be stored in a glass stoppered bottle and kept in dark and its exact normality ascertained with standard oxalic acid solution.

\*Rules for rounding off numerical values ( *revised* ).

†Specification for test sieves ( *revised* ).

‡Specification for water, distilled quality ( *revised* ).

**3.2 Oxalic Acid** — N/10; weigh 1.575 g of  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  and make up to 250 ml in a volumetric flask.

**3.3 Sulphuric Acid** — 2 N; add 12.5 ml of pure sulphuric acid to 240 ml of water.

**3.4 Ammonium Nitrate** — Dissolve 20 g of ammonium nitrate in one litre of water.

**3.5 Hydrochloric Acid** — 2 N; sp gr 1.19 mixed with equal volume of distilled water.

**3.6 Ammonium Oxalate** — saturated solution.

**3.7 Ammonium Hydroxide** — sp gr 0.89. Mix ammonium hydroxide and distilled water in the ratio of 1 : 2 (one part of ammonia and two parts of distilled water).

**3.8 Calcium Chloride Solution** — Dissolve one part by weight of salt in 100 parts by weight of distilled water.

#### 4. SAMPLES

**4.0** Samples given in 4.1 to 4.3 shall be selected for the test as given in IS : 4332 ( Part I )-1967\*.

**4.1 Raw Soil** — representative of the soil phase of the soil-cement mixture.

NOTE — The results of this test are very sensitive to the variation of calcium content in soil. Hence, due attention should be paid to sampling to obtain representative samples and the number of samples to be tested.

**4.2 Cement** — representative of the cement phase of the soil-cement mixture.

**4.3 Soil-Cement** — representative sample of the mixture to be analyzed.

#### 5. PROCEDURE

**5.1** Dry 25 g of each sample in the drying oven at  $110 \pm 5^\circ\text{C}$  to a constant weight. Pulverize the samples to pass through a 425-micron IS Sieve.

**5.2** Weigh out separately on analytical balance raw soil, 5 g; soil-cement mixture, 5 g; and cement, 1 g. Transfer each of the weighed samples to separate 250-ml beakers. Add 50 ml of hydrochloric acid (2N), to each sample. Cover it and boil gently on a hot-plate for 5 min.

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\*Methods of test for stabilized soils: Part I Sampling and preparation of stabilized soils for testing.

**5.3** To each of the beakers add 25 ml of hot distilled water and filter the contents through Whatman No. 42 filter paper and receive each filtrate in a 250-ml volumetric flask separately. The material on the filter should be washed with small amounts of hot water repeatedly till free from chloride ions (about 4 times) (check with silver nitrate solution).

**5.3.1** Take a small quantity of filtrate (say 1 cc) obtained from the soil-cement mixture treated by hydrochloric acid and add 3 cc of ammonium molybdate reagent and a few drops of concentrated nitric acid and warm to 40°C. A yellow precipitate indicates the presence of phosphate ions.

**5.4** After completion of washing, discard the filter paper and dilute the filtrate in the volumetric flask to 250 ml with cold water. Shake the contents well and remove a 50-ml aliquot and transfer to the original 250-ml beaker (see 5.2), using 250-ml pipette. Dilute to 100 ml. Add a few millilitres of bromine water or a few drops of concentrated nitric acid. Boil, cool and make the solution slightly ammoniacal with ammonium hydroxide. Boil for two minutes and allow the hydroxide to settle.

**5.4.1** In case of soil-cement mixture containing phosphate, dissolve the precipitates of hydroxides in the minimum quantity of dilute hydrochloric acid. To the main solution, add dilute ammonia solution drop by drop with stirring, until either a faint permanent precipitate is just obtained or the solution is just alkaline. Then add 2 to 3 ml of dilute acetic acid (1 : 1) and 10 ml of 3N ammonium acetate solution. Discard any precipitate, which may form at this stage. If the solution is red, sufficient ferric ion is present in the solution combined with all the phosphate ions. If the solution is not red in colour, add neutral ferric chloride ( $\text{FeCl}_3$ ) solution, drop by drop and with stirring until the solution acquires a deep brownish red colour. Dilute the solution to 150 ml with hot water, boil gently, filter hot and wash the residue with a little boiling water. The residue will contain the phosphates of Fe, Al and Cr and may be discarded. Boil down the filter to 20 to 25 cc and proceed as given in 5.6.

**5.5** Filter hydroxides except soil-cement mixture containing phosphates for which the process of separation has been given in 5.4.1 through Whatman No. 41 filter paper, receiving filtrate in a 600-ml beaker. Wash the original 250-ml beaker into the filter once with a stream of ammonium nitrate and then with hot ammonium nitrate twice. Set aside the filtrate and place original beaker under funnel. Perforate the paper with a rod and wash the hydroxides into the original beaker with hot ammonium nitrate to remove most of the precipitate from the filter paper. Treat the paper with 2 ml of hot hydrochloric acid (1 : 3). Wash the paper

several times with hot water, and then discard the paper. Dilute the solution to 75 ml.

**5.6** Make the solution slightly ammoniacal with constant stirring and boil for 1 to 2 minutes. Allow the precipitate to settle and filter through Whatman No. 41 filter paper. Receive the filtrate in 600-ml beaker ( *see* 5.5 ). Wash the precipitate with ammonium nitrate three to four times. Discard the hydroxide precipitate. Add 2 to 3 drops of methyl red indicator and ammonium hydroxide ( sp gr 0.89 ) to the filtrate till the colour changes from faint pink to yellow (  $pH \approx 5$ , optimum for calcium oxalate precipitation ). Heat the solution to boiling and add 10 ml of hot saturated ammonium oxalate solution. Keep the mixture near boiling until the precipitate becomes 'granular', then set aside on a warm hot-plate for 30 min or more. Check the completeness of precipitation. Filter off calcium oxalate precipitate through Whatman No. 42. Clean the beaker with rubber policeman and transfer the contents to the filter with a stream of hot water. Wash the filter 8 to 10 times with hot water or preferably four times each with ammonium hydroxide ( 2.98 ) to make sure that soluble oxalates are completely removed. This can be checked with calcium chloride solution

**NOTE** — Calcium content in the soil, cement and soil-cement mixture may also be determined by rapid method given below:

Filter the acid solution ( *see* 5.4 ), wash and make up to 100 ml of which 50 ml are just neutralized with liquid ammonia. Dissolve the precipitates of aluminium oxide and ferric oxide thus formed by just the quantity of glacial acetic acid required for dissolving the precipitates. Then add in excess saturated solution of ammonium oxalate to the solution to form a copious precipitate of calcium oxalate. Filter off calcium oxalate precipitate, wash with hot water till free from oxalates. Dissolve the oxalates in ( 1 : 4 ) sulphuric acid and titrate with standard potassium permanganate solution to determine the percentage of calcium.

**5.7** Open the filter paper carefully and wash the precipitate into the beaker in which the precipitation was effected. Dilute to 200 ml and add 10 ml of sulphuric acid ( 1 : 1 ).

**5.8** Heat the aliquot portion ( 5.7 ) to 65°C on a water-bath maintained at 60 to 65°C and titrate it with standard potassium permanganate solution to a persistent pink colour for 10 seconds. Take two more readings. Five times the average value is the reading *P* as in 6.1.

**5.9 Blank** — Make a blank determination, following the same procedure using same amount of reagents.

## 6. CALCULATIONS

**6.0 Cement Content of the Soil** — Cement mixture should be calculated as given in 6.1 and 6.2.

**IS : 4332 ( Part VII ) - 1973**

**6.1** Calculate the percentages of calcium oxide in the soil, the cement, and the soil-cement mixture as follows:

$$\text{Percentage of calcium oxide} = \frac{(P - Q) R \times 0.028}{S} \times 100$$

where

$P$  = ml of potassium permanganate solution required for titration of the sample;

$Q$  = ml of potassium permanganate solution required for titration of the blank;

$R$  = normality of the potassium permanganate solution = 0.1;

$S$  = weight of sample represented by the aliquot titrated, in g; and

0.028 = calcium oxide equivalent of 1 ml of 1 N potassium permanganate.

**6.2** Calculate the percentage by weight of cement in the soil-cement mixture as follows:

$$\text{Percentage cement} = \frac{(X - Y)}{Z} \times 100$$

where

$X$  = percentage of calcium oxide in soil-cement mixture,

$Y$  = percentage of calcium oxide in raw soil, and

$Z$  = percentage of calcium oxide in cement.

**NOTE** — When hydrated soil-cement mixtures ( that is, mixtures prepared and laid sometime earlier ) are analyzed, the value of percentage by weight of cement obtained is in terms of hydrated cement. Such values need to be converted to an approximate equivalent of dry cement, through a factor which is of the order of 1.04.

*( Continued from page 2 )*

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